

PDL/ACF/048/98

Aim: Preparation of ACE from 7-ACA
(Bio-chem.)

"Pre-demonstration"

Repetition 2 PDL/ACF/048/98

STAGE - I

Prepⁿ of TEA

2-Furyl chloride	: 55.0 gm
NaOH NaOH	: 60.0 gm
DMW	: 75 (730 + 950) ml
EtOAc	: (500 + 500) ml
1.1HCl	: (70 + 100) ml
NaHCO ₃	: 40.0 gm

STAGE - 2

Prepⁿ of ACE

7-ACA	: 100.0 gm
EtOAc	: 400.0 ml
BF ₃	: 137.0 gm
EDTA	: (2.0 + 0.5) gm
TEA	: 250.0 ml
DMF	: 2.0 gm / 2
DMIO	: 300.0 ml / 2

DMIO	: (300 + 100) ml
EtOAc	: 400 + (50 x 100) ml

	A	B
12V NH ₂ SM	114.0 ml	—
20x NH ₂ OH	—	183.0 ml
DMIO	(200 + 50) ml	200 + 50 ml
EtOAc	150 + 50 ml	(150 + 50) ml

1st/nd. 3 013 242.4 / 250.0ml 95.25%

1st/nd. 3 AL3 478.3 / 420ml 90.09%

R/m reported 0.14 0.25%

1st/nd. 3 4.36% 11.05% 83.09%

242.4ml 0.34% 7.77% 89.53% 0.92%

Day with 58.7 58.9

M/c

PDL/ACF/051/98

Aim: Preparation of ACF from 7-ACA (Biochemi)

Raw materials

Stage-I, Prepⁿ of TEA

o-Fumyl chloride	55.0 gm
NaSH	60.0 gm
DMW	(700 + 30 + 350) ml
EtOAc	(500 + 200) ml
1.1HCl	(70 + 100) ml
NaHCO ₃	40.0 gm
	240 ml

Vol. of TEA : 260.0 ml

Stage-II, Prepⁿ of ACF

7ACA	100.0 gm
EtOAc	400.0 ml
BE ₂	137.0 gm
TEA	260.0 ml
EDTA	(60.0 + 0.6) gm
SHS	2.0 gm
DMW	300.0 ml

13% NH₃ soln

EtOAc : 100 + 300 + 600

DMW : 300 + (2 x 100) ml

Wt at: 2.5. Fgl

PDL/ACF/053/90.

Aim: Preparation of BACE from 7-ACA (Biochemi-
 Media 120gms BE3 gas purged instead of 137g. ii)

Raw materials Stage-I

2-Ethyl chloride : 55.0gm

NaSH : 60.0gm

DMW : (130+350)ml

EtOAc : (500+200)ml

1M HCl : (70+100)ml

NaHCO₃ : 40.0gm

Stage-II

7-ACA : 100.0gm

EtOAc : 400.0ml

BE3 : 3.2A (P) : 130.0gm

TEA : 80% : 25.0ml

SHS : 2.0ml

EDTA : (2.0+0.5)gm

DMW : 300.0ml

13K N₂ : 200.0ml pH 3.5

EtOAc : (100+300+100)ml

DMW : (300+100+100)ml

No Res. Monitoring

Terminated in 3rd hr.

Wet wt : 269.0 gms.

DMW : 550 ml

BE3 : 8.25 ml

used in PDL/ACF/053/90 & 054/90

PDL/ACE/055/98

AIM: Preparation of ACE from FACA (Biochemi)

Raw materials Stage-I TFA prep?

2-Ethyl chloride

55.0 gm

NaSH

60.0 gm

DMW

(700 + 30 + 350) ml

EtOAc

(500 + 200) ml

1.1HCl

(723 + 100) ml

NaHCO₃

40.0 gm

Stage-II

ACE prep?

FACA

100.0 gm

EtOAc

400.0 ml

BF₃

137.0 gm

EDTA

(200 + 0.6) gm

TFA

255.0 ml (99.68%)

SH₃

2.0 gm

13% NH₃ soln

200.0 ml

DMW

300.0 ml

EtOAc

(100 + 300 + 100) ml

DMW

(300 + 100 + 100) ml

R/m report:

FACA

ACE

TFA

total

2 hrs 30

0.35%

85.61%

8.16%

3.49%

3 hrs

0.35%

85.50%

8.26%

3.67%

Terminated in 3 hrs

Net wt : 249.7 gm

Aim: Preparation of ACF from 7-ACA

Raw materials:

Stage - I prepⁿ of TEA

2-Furanyl chloride : 55.0 gm
 NaOH : 60.0 gm
 DMF : (700 + 30 + 350) ml
 EtOAc : (500 + 200) ml
 11 HCl : (75 + 100) ml
 NaHCO₃ : 40.0 gm

Stage - II prepⁿ of ACF

TEA : 255.0 ml (Syn. problem)
 7-ACA : 100.0 gm
 EtOAc : 400.0 ml
 BF₃ : 127.0 gm
 EDTAC : (2.0 + 0.6) gm
 SHSAC : 2.0 gm
 DMF : 300.0 ml
 13% NH₃ solⁿ : 240 ml
 EtOAc : (100 + 300 + 100) ml
 DMF : (300 + 100 + 100) ml

R/m Report	7-ACA	ACF	TEA	Imp
2 1/2 hrs	8.33	82.74	6.62	3.55
3 hrs	0.31	88.01	6.57	3.92

Terminated in 3 hrs.

Wet wt: 276.4 g.

Aim: Preparation of ACF from FACA

Raw materials

Stage-1. Prepⁿ of TEA

2-Furoyl chloride : 55.0gm
 NaSH : 60.0gm
 DMW : (100+30+350)ml
 EtOAc : (500+200)ml
 12% HCl : (75+200)ml
 NaHCO₃ : 40.0gm

Prepⁿ of ACF Stage-1

FACA : 100.0gm
 EtOAc : 400.0ml
 BF₃ : 137.0gm
 EDTA : (2.0+0.6)gm
 TEA : 255.0ml
 SHS : 2.0gm
 DMW : 200.0ml
 13% NH₃ solⁿ : 233ml
 EtOAc : (100+300+100)ml
 DMW : (300+100+100)ml

R/m. report	FACA	ACF	TEA	Imp.
2 1/2 hrs.	0.55	87.38	7.40	1.98
3 hrs.	0.47	88.78	6.92	2.08

Terminated in 3 hrs.

Net wt : 226.3 gms.

Used in PDL/ACF/157/98